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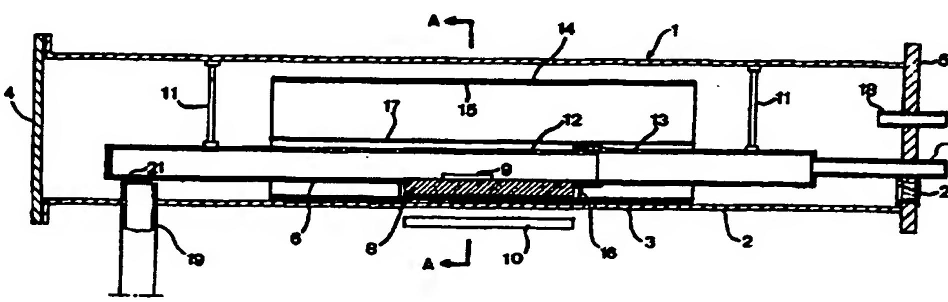
(54) Title: DEVICE FOR HEAT SHIELDING WHEN SiC IS GROWN BY CVD



(57) Abstract

A device for shielding the environment against the heat produced when SiC is epitaxially grown by Chemical Vapour Deposition on a substrate (9) by using a susceptor (8) heated for heating the substrate and a gas mixture fed to the substrate for the growth, comprises a tube defining a room (22) arranged to receive the susceptor and the substrate. The inner walls of the tube are at least close to the susceptor (8) coated by a thin heat-reflecting film (15), preferably a carbon film. This film may be applied to the inside of the tube by introducing a C-containing gas, which is heated until cracking. Enclosing the substrate and the susceptor in a casing (2, 14) and flushing the casing with Ar-gas is another way of shielding the environment against the heat produced by the process.

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Device for heat shielding when SiC is grown by CVD

TECHNICAL FIELD OF THE INVENTION AND PRIOR ART

The present invention relates to a device for shielding the environment against the heat produced when SiC is epitaxially grown by Chemical Vapour Deposition on a substrate while using a susceptor heated for heating the substrate and a gas mixture fed to the substrate for the growth, said device comprising a tube defining a room arranged to receive the susceptor and the substrate.

SiC single crystals are in particular grown for being used in different types of semiconductor devices, such as for example different types of diodes, transistors and thyristors, which are intended for applications in which it is possible to benefit from the superior properties of SiC in comparison with especially Si, namely the capability of SiC to function well under extreme conditions. SiC has a high thermal stability due to a large band gap between the valence band and the conduction band, such that devices fabricated from said material are able to operate at high temperatures, namely up to 1000K.

However, the high thermal stability of SiC also means that high temperatures are needed for obtaining a good ordered growth thereof. The epitaxial growth of silicon carbide by Chemical Vapour Deposition is therefore carried out in a temperature regime in excess of 1400°C. These high temperatures are needed both to obtain decomposition by cracking of the Si- and C-containing precursor gases of said gas mixture and to ensure that the atoms are deposited on the substrate surface in a good order. These high temperatures in a reactor for Chemical Vapour Deposition of SiC also means problems with shielding the environment, primarily the walls

of a vacuum casing in which the growth takes place and other surrounding equipment, against the heat emanating from the susceptor. For this reason a tube as defined in the introduction has been arranged, and this tube has been made of graphite felt, which absorbs and retain the heat emitted from the susceptor, so that less heat has to be supplied to the susceptor and the reactor casing and other environmental equipment do not get too hot.

The graphite felt is selected as material for the tube since it is comparatively free from impurities, it is easily available, it shields the heat very well and it is permeable to Rf-field radiation, which is of importance, since the susceptor is mostly heated by a Rf-coil located outside the reactor casing.

However, the graphite felt is porous and it may raise dust because particles thereof may be torn away and if they land on the substrate they will cause severe defects of the crystal grown thereon, and said felt does also attract impurities from said gas mixture which may later on be released and incorporated into the epitaxially layers of the SiC crystal grown and by that cause so called "memory effects" thereof.

SUMMARY OF THE INVENTION

The object of the present invention is to provide a device as defined in the introduction, which makes it possible to shield the environment against said heat just as well or even better than when using a tube of graphite felt in such a device, but through which a remedy is find to the inconveniences associated with the use of graphite felt as material for said tube.

This object is in accordance with the invention obtained by providing said device with a tube having the inner walls at least close to the susceptor coated by a thin heat-reflecting film. It has been found that such a thin heat-reflecting film will reflect the heat coming from the susceptor back in the direction of the susceptor, so that it may be used for heating the substrate and said gas mixture and less

heat causing problems with cooling has to be supplied to the susceptor, which means that a surrounding casing and other equipment does not get too hot. Furthermore, it has surprisingly turned out that the temperature drop through such a thin heat-reflecting film will be considerable, so that materials which are preferred to be used in reactors of this type, but which do not withstand the high temperatures needed for the substrate and cracking of the gases of said gas mixture, may be used as materials for said tube. Furthermore, and this is very important, such a film will not raise any dust as the graphite felt. A further advantage of such a film is that it does not absorb impurities causing so called "memory effects" from the gas mixture, so that the defects in the epitaxially grown SiC-layers may be reduced considerably.

According to a preferred embodiment of the invention said film is a carbon film. It has been found that a thin carbon film is well suited as the thin heat-reflecting film with the object of the invention. A carbon film reflects the heat coming from the susceptor very efficiently, so that the temperature on the external side of the film will be several hundred °C lower than the temperature of the susceptor.

According to another preferred embodiment of the invention said tube is made of quarts internally coated by said film. Thanks to the use of said thin film quartz may be used as material for the tube, which is a desired material for the tube, since quartz may be easily flushed free from impurities by flushing gases and is also permeable to Rf-field radiation, so that Rf-filed radiating means may be used for heating the susceptor. Quartz can not stand temperatures above 1300°C, which should be compared with temperatures of 1500-1700°C mostly used for said epitaxial growth, but thanks to the application of said thin film, the temperature of the tube may be held below that upper limit for quartz. It has been found that the application of a carbon film on the tube of quartz will result in a temperature of only 1250°C at the external side of the film, when the susceptor temperature is 1550°C.

According to a still further preferred embodiment of the invention said film has a thickness of 2-10 μm. A film, preferably a carbon film, having a thickness within this interval has turned out to be most suitable, since an even thinner film will not reflect that good and a thicker film will be pealed off from the tube.

The carbon film may in accordance with the invention be applied to the inside of a tube to be used for defining a room arranged to receive a susceptor and a substrate in a device for epitaxially growing SiC by Chemical Vapour Deposition by introducing a C-containing gas into the tube and heating said gas until cracking thereof for deposition of carbon atoms as an even layer on the internal wall of the tube. This method gives rise to a very even black metal-like high-reflecting layer of carbon on the inside of the tube. It is advantageous to use methane gas as the C-containing gas.

Another object of the invention is to provide a method for shielding the environment against the heat produced when SiC is epitaxially grown by Chemical Vapour Deposition on a substrate while using a susceptor heated for heating the substrate and a gas mixture fed to the substrate for the growth, said substrate and susceptor being enclosed in a casing. This object is in accordance with the invention obtained by flushing the casing with Ar-gas acting as a shield against transferral of heat from said susceptor to the casing through the gas located therebetween. Thus, by using argon gas as flushing gas when SiC is grown by Chemical Vapour Deposition the very low thermal conductivity of argon leads to a better protecting of primarily the casing material than in the prior art methods, which have primarily used hydrogen gas as flushing gas. Thus, the heat transferral through the gas is drastically reduced, but the heat radiation from the susceptor is of course not influenced. Furthermore, it would not be critical if argon would reach the vicinity of the substrate, since it will not be incorporated as impurity in the epitaxial layer of the SiC crystal.

Further advantages and preferred characteristics of the invention will appear from the following description and the other dependent claims.

BRIEF DESCRIPTION OF THE DRAWING

With reference to the appended drawing, below follows a specific description of a preferred embodiment of the invention cited as an example.

In the drawing:

Fig 1 is a simplified longitudinal cross-section view of a reactor for epitaxial growth of SiC by Chemical Vapour Deposition including a device according to a preferred embodiment of the invention and

Fig 2 is a cross-section according to A-A in Fig 1 of the reactor according to Fig 1, in which some further unimportant details are omitted.

DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT OF THE INVENTION

A reactor 1 for epitaxially growing SiC by Chemical Vapour Deposition is schematically shown in Fig 1 for explaining the principals of the invention. The reactor 1 comprises a casing 2 constituted by an outer tube 3 of quartz and end flanges 4 and 5 of stainless steel. The casing 2 delimits an inner volume in which vacuum is created by pumps not shown. The reactor further comprises an inner cell 6 of graphite in the form of a tube extending in the longitudinal direction of the outer tube 3. A gas mixture of C- and Si-containing gases and a H_2 carrier gas will be fed to the inner cell 6 through a conduit 7 penetrating the end flange 5.

The inner cell 6 has a rectangular cross-section with a comparatively small height, as seen in Fig 2, and a part of the bottom of the inner cell is formed by a susceptor 8 of graphite hanging in the inner

cell. The susceptor 8 is arranged to receive a substrate 9 thereon for epitaxial growth of layers of SiC thereon. The substrate 9 may for instance be crystalline SiC, Si or any Group III-nitride. The reactor does also comprise a heating means 10 in the form of a Rf-field radiating coil arranged outside the casing 2 in the region of the susceptor and arranged to heat the susceptor by the Rf-field radiated thereby. Thanks to the fact that the casing 2 is made of quartz, it will be permeable to said Rf-field. The susceptor 8 is furthermore preferably coated by SiC, at least close to the substrate 9, for preventing impurities in the graphite of the susceptor from being released into the inner cell and be incorporated into the epitaxial layers of the substrate. The inner cell surfaces close to the susceptor are preferably also coated by SiC for the same reason.

The inner cell 6 is suspended in the casing by rods 11 of quartz or aluminium oxide, and it is made of two parts 12, 13 connectable to and removable from each other, so that the part 12 containing the susceptor (to the left in Fig 1) may be removed from the casing 2 after removal of the end flange 4 for checking the SiC crystal grown, replacing said SiC crystal by a new substrate, checking the state of the susceptor and so on. A susceptor may then also be removed from the inner cell for replacement or conditioning.

The reactor also comprises a tube 14 of quartz with a somewhat smaller diameter than the outer tube 3 and which in a room 22 defined thereby receives the inner cell and the susceptor. The inner tube 14 bears on the bottom of the outer tube 3. The tube 14 is internally coated by a thin carbon film 15 with a thickness of 2-10 μ m. This carbon film is highly heat-reflecting and have a metal-like lustrous character. It is created by introducing a C-containing gas, preferably methane gas, into the tube and heating said gas at a temperature of 1150-1250°C, so that the molecules of the gas are cracked and carbon atoms are deposited as an even layer on the internal wall of the tube. This is accordingly a process for manufacturing the tube 14. It has been found that the C-containing gas does hardly crack at a lower heating temperature than 1150°C and the quartz may be damaged if the temperature exceeds 1250°C. Thanks

to the fact that the tube is made of quartz and the carbon film is thin, it will be permeable to the Rf-field emitted by the heating means 10.

The reactor also comprises two graphite plates 16 arranged on both sides of the susceptor 8 for reflecting heat radiated by the susceptor back towards the susceptor as well as a graphite plate 17 deviding the inner tube 14 into two parts and arranged to reflect heat coming primarily from the susceptor and the walls of the inner cell back.

The reactor also comprises means indicated at 18 for supplying a flushing stream of Ar-gas to the casing, which is intended to flow through the casing and leave this in a tube 19 connected to a pump not shown. The Ar-gas is intended to flush the walls of the inner 16 and outer 3 tube of quartz for removing impurities thereon. The main reason for choosing quartz as a material for these tubes is exactly that impurities deposited thereon may very easily be flushed away. It has also been found that the Ar-gas has a not neglectible heat shielding effect, since argon has a very low thermal conductivity. More precisely, the argon gas flows in a small gap 23 between the susceptor and the inner tube 14 and reduces the heat transferral from the susceptor to the inner surface of the tube 14 through the gas therebetween. The argon gas also reduces such heat transferral to other parts of the tube 14.

The reactor has also a window 20 making it possible to determine the temperature in the susceptor 8 by looking at a bore drilled in the susceptor and measuring the radiation from such an "ideal" black body.

The reactor is used to grow films with a thickness of 1-50 μm for the use in primarily high power semiconductor devices and is as follows: a gas mixture of C- and Si-containing precursor gases, for instances propane and silane, and a carrier gas, preferably H_2, is introduced into the conduit 7 and drawn through the inner cell 6. A diffuser means not shown is arranged in the transition between the conduit 7 and the inner cell 6 so as to prevent the formation of a

central jet inside the inner cell 6. The heating means 10 heats the susceptor 8 and by that the substrate 9 and the gas mixture passing closely above the susceptor and the substrate to a temperature of preferably 1500-1600°C, so that the precursor gases are cracked and the Si- and C-atoms so formed are deposited onto the substrate 9 while forming well ordered epitaxial layers thereon. The substrate 9 is placed downstreams of the longitudinal middle of the susceptor 8, so that the precursor gases of the gas mixture will reach a temperature resulting in cracking thereof before or when they reach the region of the substrate 9. The comparatively low height of the inner cell 6 make the gases pass closely above the susceptor and the substrate and facilitates the heating of the gas mixture, so that the epitaxial growth of SiC on the substrate 9 is promoted.

The heat radiated by the susceptor 8 will reach the tube 14, which in particularly close to the susceptor, namely in the region thereunder, will receive comparatively much thermal energy per time- and surface-unit. However, the carbon film 15 of the tube 14 will reflect said heat back towards the susceptor and the inner cell, so that there will be a temperature fall through said film 15 and it will on the outer side thereof only have a temperature of less than 1250°C, which will be of no problem to the quartz material of the tube 14. Thus, the arrangement of said thin carbon film will result in a lower heat supply from the heating means 10 thanks to the reflection of the heat back towards the regions of the inner cell to be heated, so that the temperature of the tube 14 and the casing 2 will be lower than otherwise without any need of any cooling by cooling fluids or the like. This does also make it possible to use quartz as material for the tube 14 and for the casing 2 without any need of additional cooling means.

The carrier gas of the gas mixture and remaining parts of the precursor gases and products of the cracking thereof will leave the inner cell through an opening 21 thereof near its end and through the tube 19. There is a small gap, which has been exaggerated in Fig 1, between the opening 21 and the tube 19, so that the flushing gas introduced through the conduit 18 and possibly other conduits

not shown may also leave the casing 2 through the tube 19. Apart from this opening 21 the inner cell 6 will be sealed with respect to the rest of the casing.

The invention is of course not in any way restricted to the preferred embodiment described above, but several possibilities to modifications thereof would be apparent to a man with ordinary skill in the art without departing from the basic idea of the invention.

The heat shielding tube may be made of another material than quartz onto which a thin heat-reflecting film will be possible to be deposited, and this material may also be impermeable to Rf-field radiation, although that will complicate the heating of the susceptor.

All parts of the reactor shown in the figures may of course have other geometric forms, and "tube" as defined in the claims has to be interpreted in its broadest sense also including for instance rectangular, triangular and other cross-section shapes.

Although it has been shown how the entire heat shielding tube is internally coated by the thin heat-reflecting film, it will be possible to coat only portions thereof with such a film, which portions should at least comprise the portions close to the susceptor.

Furthermore, said heat shielding tube does not have to extend so far upstreams and downstreams of the susceptor as shown in Fig 1, but the extension thereof may be adapted to the actual conditions in the reactor in question.

"The environment" in the claims has to be interpreted very broadly and comprises all types of equipment, walls and the like arranged near the susceptor and which by that may be exerted to heat deriving therefrom.

The definition "casing" in the claims includes all types of elements having walls enclosing the susceptor and the substrate, such as the inner tube described above.

Claims

1. A device for shielding the environment against the heat produced when SiC is epitaxially grown by Chemical Vapour Deposition on a substrate (9) while using a susceptor (8) heated for heating the substrate and a gas mixture fed to the substrate for the growth, said device comprising a tube (14) defining a room arranged to receive the susceptor and the substrate,
characterized in that the inner walls of the tube (14) are at least close to the susceptor coated by a thin heat-reflecting film (15).

2. A device according to claim 1,
characterized in that said film (15) is a carbon film.

3. A device according to claim 1 or 2, in which the susceptor (8) is heated by Rf-field radiating means (10) located outside said tube (14),
characterized in that the tube is made of a material permeable to said Rf-field internally coated by said film (15).

4. A device according to any of claims 1-3,
characterized in that said tube (14) is made of quartz internally coated by said film (15).

5. A device according to any of claims 1-4,
characterized in that the film (15) has a thickness of 2-10 μm.

6. A device according to any of claims 1-5,
characterized in that said film (15) has a metal-like lustrous character.

7. A method for applying a thin carbon film (15) to the inside of a tube (14) to be used for defining a room (22) arranged to receive a susceptor and a substrate in a device for epitaxially growing SiC by Chemical Vapour Deposition,

characterized in that a C-containing gas is introduced into the tube (14) and heated until cracking thereof for deposition of carbon atoms as an even layer on the internal wall of the tube.

8. A method according to claim 7, characterized in that the C-containing gas is methane gas.

9. A method according to claim 8 or 9, characterized in that said heating is carried out at a temperature of 1150-1250°C.

10. A method according to any of claims 7-9, characterized in that the carbon atoms are deposited on a tube (14) of quartz.

11. A method for shielding the environment against the heat produced when SiC is epitaxially grown by Chemical Vapour Deposition on a substrate (9) while using a susceptor (8) heated for heating the substrate and a gas mixture fed to the substrate for the growth, said substrate and susceptor being enclosed in a casing (2, 14), characterized in that the casing is flushed with Ar-gas acting as a shield against transferral of heat from said susceptor (8) to the casing (2, 14) through the gas located therebetween.

12. A method according to claim 11, characterized in that said Ar-gas is at least led between the hottest surfaces of the susceptor (8) and the surrounding casing (14) material.

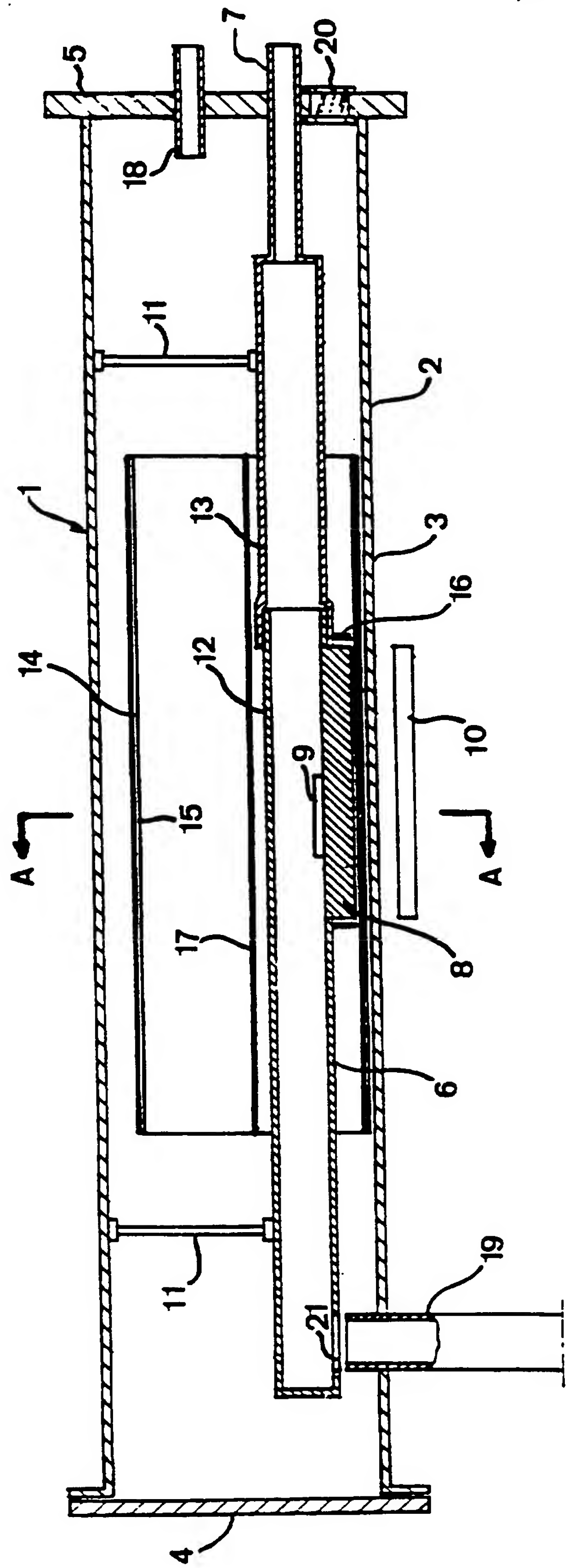
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Fig 1

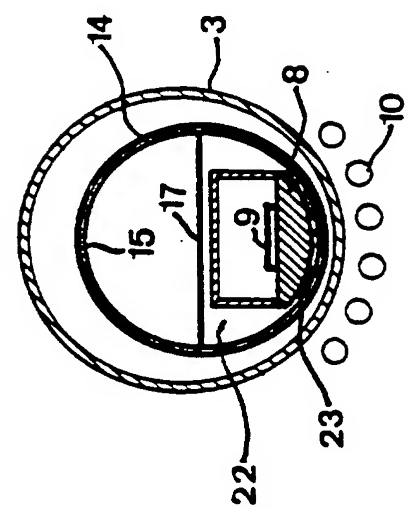
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Fig 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 96/00070

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C30B 29/36, C30B 29/08, C30B 29/02, C23C 16/32
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C30B, C23C, H01L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, JAPIO

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0147967 A2 (APPLIED MATERIALS, INC.), 10 July 1985 (10.07.85), page 2, line 30 - page 3, line 13; page 19, line 10 - page 24, line 32, figure 8, claim 3 --	1-6
Y	EP 0269439 A2 (SHARP KABUSHIKI KAISHA), 1 June 1988 (01.06.88), column 5, line 10 - line 56, figure 2, claim 3 --	1,3,4
Y	US 3845738 A (SAMUEL BERKMAN ET AL), 5 November 1974 (05.11.74), column 3, line 38 - line 40; column 4, line 16 - line 19, abstract --	2-6

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" ertier document but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search 14 May 1996	Date of mailing of the international search report 15 -05- 1996
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Ingrid Grundfelt Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 96/00070

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 1134133 A (QUARTZ AND SILICE), 20 November 1968 (20.11.68), page 3, line 34 - line 45, claim 1	7-10
	--	
X	EP 0045192 A2 (MONSANTO COMPANY), 3 February 1982 (03.02.82), claims 1,2	11,12
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A	EP 0446988 A1 (ENIRICERCHE S.P.A.), 18 Sept 1991 (18.09.91), column 3, line 45 - line 51	11,12
	--	

INTERNATIONAL SEARCH REPORT

International application No.
SE/96/00070

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

Claims 1-10 relate to a device and to a method to be used when SiC is epitaxially grown by CVD. "The special technical feature" of claims 1-10 relates to a thin heat-reflecting film to be applied to the inside of the reactor tube.

Claims 11-12 relate to a method to be used when SiC is epitaxially grown by CVD. "The special technical feature" of claims 11-12 relates to an argon-flushing of the reactor casing.

These groups of inventions are not linked so as to form a single inventive concept.

There is no technical relationship between those inventions involving one or more of the same or corresponding technical features.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT
Information on patent family members

01/04/96

International application No.
PCT/SE 96/00070

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A2- 0147967	10/07/85	DE-A- 3485898	01/10/92
		JP-A- 60186013	21/09/85
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		JP-C- 1661575	19/05/92
		JP-B- 3027515	16/04/91
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		JP-A- 63136511	08/06/88
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		US-A- 4309241	05/01/82
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		IT-D,V- 1962790	15/11/90
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		US-A- 5141613	25/08/92